भारतीय मानक

एसीसलफेम पोटाशियम, खाद्य ग्रेड — विशिष्टि

Indian Standard

ACESULFAME POTASSIUM, FOOD GRADE — SPECIFICATION

ICS 67.220.20

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Food Additives Sectional Committee, FAD 8

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

Acesulfame potassium is a sweetener and flavour enhancer. Acesulfame K is 180-200 times sweeter than sucrose (table sugar), as sweet as aspartame, about half as sweet as saccharin, and one-quarter as sweet as sucralose. Acesulfame K is often blended with other sweeteners (usually sucralose or aspartame). These blends are reputed to give a more sugar-like taste whereby each sweetener masks the other's after taste, and/or exhibits a synergistic effect by which the blend is sweeter than its components. Unlike aspartame, acesulfame K is stable under heat, even under moderately acidic or basic conditions, allowing it to be used in baking, or in products that require a long shelf life.

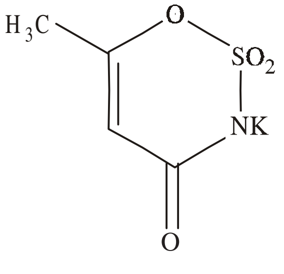
To ensure the quality of the product, it is necessary to have quality control specifications. While formulating this standard, considerable assistance has been derived from JECFA standard for Pectin, published in FNP 52 Add 9 (2001). This standard has been formulated to provide the necessary guidelines to manufacturers and the consumers for maintaining quality control.

In the formulation of this standard, due consideration has been given to the *Food Safety and Standards Act*, 2006 and the Rules framed thereunder. Due consideration has also been given to the *legal Metrology (Packaged Commodities) Rules*, 2011. However, this standard is subject to restrictions imposed under these, wherever applicable.

Description

Chemical names	—	Potassium salt of 6-methyl-1,2,3-oxathiazine-4(3H)-one-2,2-dioxide Potassium salt of 3,4-dihydro-6-methyl-1,2,3-oxathiazine-4-one-2,2-dioxide
C.A.S. number	—	55589-62-3
Chemical formula	—	$C_4H_4KNO_4S$
Formula weight	—	201.24
Solubility	—	Freely soluble in water, very slightly soluble in ethanol.

Structural formula:



In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated expressing the result of test or analysis, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

ACESULFAME POTASSIUM, FOOD GRADE — SPECIFICATION

1 SCOPE

This standard prescribes requirements, methods of sampling and test for acesulfame potassium, food grade.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1699 : 1995	Methods of sampling and test for food colours (*second revision*)
2491 : 1998	Food hygiene — General principles — Code of practice (*second revision*)

3 REQUIREMENTS

3.1 Description

Acesulfame potassium is available as an odourless, white crystalline powder.

3.2 Spectrophotometry

Dissolve 10 mg of the sample in 1 000 ml of water. The solution shows an absorbance maximum at 227 ± 2 nm.

3.3 Test for Potassium

The residue obtained by igniting 2 g of the sample should pass the test when tested in accordance with Annex A.

3.4 Precipitation Test

Add few drops of a 10 percent solution of sodium cobalt nitrite to a solution of 0.2 g of the sample in 2 ml of acetic acid TS and 2 ml of water. A yellow precipitate is produced.

3.5 Organic Impurities

It should pass the test for 20 mg/kg of UV active components when tested in accordance with Annex B.

3.6 The material shall also conform to the requirements given in Table 1.

3.7 The material shall be processed, packed, stored and distributed under hygienic conditions in licensed premises (*see* IS 2491).

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be securely packed in well-filled containers with minimum access to air. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

4.3 Marking

Each container shall be legibly and indelibly marked with the following information:

a) Name of the material including the words 'Food Grade';
b) Name and address of the manufacturer;
c) Batch or Code number;
d) Net quantity when packed;

Table 1 Requirements for Acesulfame Potassium, Food Grade

(*Clause* 3.6)

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**	
			IS No.	Annex of this Standard
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass on dry basis	99.0-100.0	—	C
ii)	Loss on drying, percent by mass, *Max*	1.0	—	D
iii)	*p*H of 1 percent solution	5.5-7.5	—	E
iv)	Fluoride, mg/kg, *Max*	3	—	F
v)	Lead (as Pb), mg/kg, *Max*	1	Clause 15 of IS 1699	—

- e) Instruction for storage;
- f) Best before date (month and year to be given by the manufacturer); and
- g) Any other requirements as given under the *Legal Metrology (Packaged Commodities) Rules,* 2011 and *Food Safety and Standards (Packaging and Labelling) Regulations*, 2011.

4.3.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

4.3.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

Representatives samples of the material shall be drawn according to the method prescribed in **4** of IS 1699.

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the experimental results or analysis.

ANNEX A

(*Clause* 3.3)

TEST FOR POTASSIUM

A-1 PROCEDURE

Potassium compounds impart a violet colour to a non-luminous flame if not masked by the presence of small quantities of sodium. In neutral, concentrated or moderately concentrated solutions of potassium salts, sodium bitartrate TS slowly produces a white, crystalline precipitate which is soluble in ammonia TS and in solutions of alkali hydroxides or carbonates. The precipitation may be accelerated by stirring or rubbing the inside of the test tube with a glass rod or by the addition of a small amount of glacial acetic acid or ethanol.

ANNEX B

(*Clause* 3.5)

TEST FOR ORGANIC IMPURITIES

B-1 APPARATUS

High performance liquid chromatography.

B-2 PROCEDURE

Perform high performance liquid chromatography using the following conditions and using 4-hydroxybenzoic acid ethyl ester as the reference substance:

- a) Column : 25 cm × 4.6 mm stainless steel
- b) Stationary phase : Reversed phase (C18 silica gel, 3-5 µm)
- c) Elution : Isocratic
- d) Mobile phase : Acetonitrile/0.01 mol/l tetrabutyl ammonium hydrogen sulphate (TBAHS) in water; 40/60 *v*/*v*
- e) Flow : About 1 ml/min
- f) Detector type : UV or Diode array, 227 nm
- g) Sample size : 20 µl of a 10 g/l solution of the sample in deionized water

NOTE — The chromatographic system must be capable of separating acesulfame K and 4-hydroxybenzoic acid ethyl ester with a resolution of 2. If peaks other than that due to acesulfame K appear within three times the elution time of acesulfame K, carry out a second run using 20 µl of a 0.2 mg/l solution of the sample. The sum of the areas of all peaks eluted in the first run within 3 times the elution time of acesulfame K elution time, except for the acesulfame K peak, does not exceed the peak area of acesulfame K in the second run.

ANNEX C

[*Table* 1, *Sl No.* (i)]

ASSAY OF ACESULFAME POTASSIUM

C-1 PROCEDURE

Dissolve about 0.15 g of the dried sample (dissolution may be slow), accurately weighed, in 50.0 ml glacial acetic acid and titrate potentiometrically with 0.1 N perchloric acid, or add two drops of crystal violet TS and titrate with 0.1 N perchloric acid, to a blue-green end-point which persists for at least 30 s. Perform a blank determination and make any necessary correction. Each ml of 0.1 N perchloric acid is equivalent to 20.12 mg of $C_4H_4KNO_4S$.

ANNEX D

[*Table* 1, *Sl No.* (ii)]

TEST FOR LOSS ON DRYING

D-1 PROCEDURE

Mix the sample well and accurately weigh 1 g of the substance. Reduce the sample to a fine powder. Tare a glass-stoppered, shallow weighing bottle that has been dried for 30 min at 105°C for 2 h. Transfer the sample into the bottle, replace the cover, and weigh the bottle and the sample. Distribute the sample as evenly as practicable to a depth of about 5 mm. Place the bottle with its contents in the drying chamber, removing the stopper and leaving it also in the chamber and dry the sample at 105°C for 2 h. Upon opening the chamber, close the bottle promptly and allow it to come to room temperature in a desiccator before weighing.

NOTE — Suitable precautionary steps should be taken when weighing hygroscopic or deliquescent samples to ensure that they do not absorb moisture.

ANNEX E

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF *p*H

E-1 APPARATUS

*p*H meter.

E-2 PROCEDURE

Each time the electrodes are used, rinse them with distilled or deionized water and carefully blot them dry with clean absorbent tissue. Form a fresh reference electrode liquid junction. Rinse the sample vessel three times with each new solution to be introduced.

Choose two standard buffers to bracket, if possible, the anticipated *p*H of the unknown. Warm or cool these standards as necessary to match within 2°C of the temperature of the unknown, and initially set the temperature compensator to that temperature. Immerse the electrodes in a portion of the first standard buffer, and following the manufacturer's instructions adjust the appropriate standardization control (knob, switch, or button) until the *p*H reading is that of the buffer. Repeat this procedure with fresh portions of the first standard buffer until two successive readings are within

± 0.02 *p*H unit without an adjustment of the standardization control.

Rinse the electrodes, blot dry, and immerse them in a portion of the second standard buffer of lower *p*H. Do not change the setting of the standardization control. Following the manufacturer's instructions, adjust the slope control (thumbwheel switch, knob, or temperature compensator) until the exact buffer *p*H is displayed. Repeat the sequence of standardization with both buffers until the *p*H readings are within ± 0.02 *p*H unit for both buffers without any adjustment of either control (the amount of sample to be used in sample preparation is given where applicable in the individual specification).

Measure the *p*H of 1 percent solution of the sample in deionized water. Always re-standardize the instrument after even a short period during which the amplifier is turned off.

NOTE — The difference between the results of two *p*H determinations when carried out simultaneously on in rapid succession by the same analyst, under the same conditions, should not exceed 0.05 *p*H unit.

ANNEX F

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF FLUORIDE

F-1 APPARATUS

F-1.1 Assembly as shown in Fig. 1.

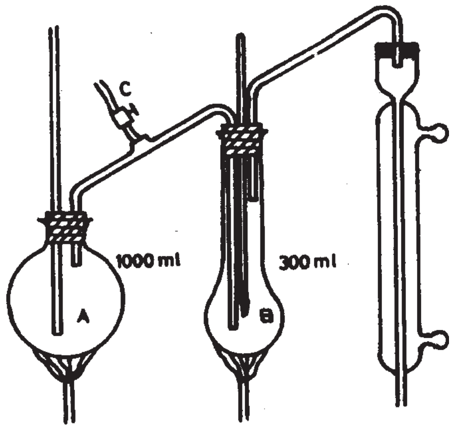


FIG. 1 APPARATUS FOR LIMIT TEST FOR FLUORIDE

F-2 REAGENTS

F-2.1 Phenolphthalein Solution — Dissolve 0.2 g of phenolphthalein in 60 ml of 90 percent ethanol and sufficient water to make 100 ml.

F-2.2 Sodium Hydroxide — 1N.

F-2.3 Sulphuric Acid — Concentrated.

F-2.4 Sodium Fluoride Solution — Containing 50 µg of fluoride.

F-2.5 Hydrochloric Acid — 4 N.

F-2.6 Zirconium Alizarin Solution — Dissolve 0.80µg of zirconium nitrate in water, add a few drops of 4N nitric acid and make up to 100 ml with water. Dissolve 0.10 g of alizarin sulphonate monohydrate in 20 ml of water, make up to 100 ml with ethanol. Mix 1 ml of first solution with 1 ml of second solution and add 18 ml of water. The solution should be clear and the dilution should be freshly prepared.

F-3 PROCEDURE

F-3.1 Place about 500 ml of water in flask *A* (*see* Fig. 1), make it alkaline to phenolphthalein solution with 1N sodium hydroxide and heat the water to boiling. Leave open the screw clamp at *C*.

F-3.2 Determine the appropriate weight of the sample (W, in g) by the formula $W = 50/L$, in which L is the fluoride limit, in mg/kg. Accurately weigh the calculated amount of the sample, place it in flask *B*, and add 10 ml of water to the flask. Add 17 ml of sulphuric acid slowly down the sides of the flask so that it forms a layer under the water. Connect flask *B* to the apparatus. Place the tip of the condenser into a flask containing 5 ml of water. Mix the contents of flask *B*, heat to 150°C and slowly shut the screw clamp at *C*. Regulate the temperature of the solution in the flask *B* to 150-153°C during the distillation. Continue until 70 ml of distillate have been collected.

F-3.3 Place the distillate in a 100 ml Nessler tube. Place 80 ml of sodium fluoride solution, containing 50 µg of fluorine, in a second Nessler tube. To each tube add 8.5 ml of 4N hydrochloric acid and 2.0 ml of zirconium-alizarin solution and make up to 100 ml with water. Let the tubes stand for 15 min.

F-3.4 The colour of the test solution containing the sample shall not be darker than that of the standard solution.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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